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Subject Additional Leaching Experiments on the Recovery of Uranium from Graphite

By H. K. Jackson

To M. McArdle

File C. F.

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M. McArdle

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[redacted]

MONSANTO CHEMICAL COMPANY
CLINTON LABORATORIES

DATE July 8th, 1947

TO M. McArdle DEPARTMENT

FROM H. K. Jackson DEPARTMENT

IN RE: Additional Leaching Experiments on the Recovery of Uranium from Graphite

Ted Davis 1/3/95

Certain experiments on the leaching of ignited graphite residues were outlined in my memo dated June 30, 1947. These experiments were designed mainly to determine preliminary rate data, and performance data on stainless steel filters.

In addition to the experiments outlined in the memorandum cited above, certain experiments should be made on the recovery of uranium from graphite by simple leaching, or grinding and leaching, without burning. The objectives of such a process might be:

1. Adequate recovery of uranium (say >99.95%) without necessity for burning.

2. Preliminary recovery of 90-95% of the uranium by a leach of the fuel units before burning. This step might be desireable for three reasons:

   A. Reduce losses which might be found inherent in the burning step.

   B. Reduce losses in the burning step occasioned by difficulties of remote control operation.

   C. Desireability of reducing activity during burning and the subsequent leaching steps. There are no data to indicate the extent to which the fission products will be leached out of the graphite. Reduction of activity by a factor of ten would probably not be significant, but a reduction by a factor of say $(10)^3$ would make a preliminary leaching attractive.

In order to allow decay of 27 formed in the pile, a cooling time of about four months will be necessary. If a preliminary leaching of the fuel units is found to be desireable, there is no reason why a leaching of 2 or 3 months might not be made.

It is known that silica has a great affinity for uranium, and it has been presumed that titanium and vanadium also retain uranium in an insoluble form. In order to increase the consistency of data, it is suggested that only Kendall type graphite be used. This type of graphite has the lowest ash content of the three types of AGOT graphite (Kendall, Gulf Clenes and Whiting).

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All references listed in MonT-320 and Memo, H.K.Jackson to File, June 30, 1947, subject: "References Pertaining to Recovery of Uranium from Graphite", are reviewed below, in so much as the references deal with the recovery of uranium by leaching without ignition.

LITERATURE REVIEW

1. Experiments by Susano and Goldberg, "Recovery of Tuballoy in Carbons", CD-GS-13, January 24, 1945.

A 25 gram sample of carbon (presumably ground), containing 5% uranium, was refluxed with 100 ml 1:1 nitric acid for a period of two hours. After the first few minutes of the period or when the reaction was running smoothly, 17 ml of hydrogen peroxide were added. After the two hour period the material was filtered and washed with 1% nitric acid, the filter paper and its contents being returned to the reflux flask for another refluxing period under the same identical conditions. After the first refluxing and washing, the recovery was 98.62%. After a second cycle (refluxing & washing) the recovery was 98.83%, and after a third cycle, the total recovery was 98.88%. The residue after the third cycle was then ignited, and the residue boiled in 20 ml of nitric acid for 25 minutes and digested for one hour. The total loss of uranium to the insoluble ash at the end of this treatment was .001% of the original uranium in the graphite.

Total weight of residue was not given.

2. Experiments by Crane, Newman and Susano, "Recovery of Tuballoy from Carbon", CD-F-4, March 8, 1945.

The authors investigated the chemical recovery of uranium from powdered carbons containing 20-40% incombustible metallic ash. Treatment with boiling dilute nitric acid under various conditions gave 85-95% recovery. Best results were obtained with two successive leaches, each with added hydrogen peroxide. It was found that incomplete ignition or "roasting" of the sample prior to leaching increased the recovery to as high as 99.95%.

Practically no details of the leaching were noted, but it was stated that early experiments indicated that extension of time beyond 30 minutes was not helpful because of formation of colloidal carbon.

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3. M-2132, August 31, 1945 (Johns Hopkins University)

A very considerable amount of work was done at Johns Hopkins University on the crushing and grinding of graphite. A high speed impact hammer mill was investigated for plant scale grinding, and tests indicated satisfactory pulverization with dust losses no greater than 0.1%. It was felt that a suitable modification of the machine would solve the problem of grinding with losses not exceeding .02 to .04%. Work on various types of mills is described.

Nitric acid extractions were made from graphite ground to pass a 40 mesh screen. Ash was 0.47% and uranium was 0.24%. 100 grams of graphite was placed in a flask equipped with reflux condenser, and boiled with 200 ml of 3 N. HNO_3. It was found possible to extract 86 - 89% by a single leach of 3 hours. Increasing the time to 6 hours did not significantly increase the amount of T extracted, "as might have been expected from the ability of the graphite to absorb leach solution." It was not found possible to extract more than 50% of the uranium from ground graphite with either carbonate or bicarbonate solutions. Addition of sodium hypochlorite to such solutions, however, resulted in 90% extraction from ground graphite in a single leach, and hence compared favorably with nitric acid.

4. M-2103, April 1, 1945 (Purdue University)

Carbon containing about 0.17% uranium was ground to below 65 mesh and extracted with nitric acid. The recovery of uranium was less than 99.9%. No other details were reported.

5. M-2105, May 1945 (Purdue University)

Carbon parts containing 0.16 uranium were ground so that all particles passed a 65 mesh sieve. This carbon was used in the following described experiments.

Three experiments were conducted in which ground carbon parts were extracted with nitric acid having concentrations of 10%, 40% and 70%. Ten grams of ground carbon parts were mixed with 100 ml of nitric acid in a round-bottom flask and the mixture was refluxed for one hour. After cooling, the carbon was separated from the nitric acid by filtration, and washed five times with conc HNO_3. In all three cases, it was found that the filtrate contained 92% of the total uranium, there being no essential difference in the recovery by extraction with 10%, 40% and 70% nitric acid.

In an experiment to determine the effect of temperature, the uranium was extracted with 50% acid for three hours. While 91% was extracted at 110°, 80% was extracted at 85°C and 62% was extracted at 30°C.

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6. A-2703, Nov. 5, 1945 (Purdue University. Summary Report on the Recovery of Uranium from Carbon Parts)

Experiments indicated that uranium is more easily and more completely extracted when the particle size is small. The data given was not on a quantitatively consistent basis.

Experiments on ground carbon parts containing 0.1% uranium indicated that 70% nitric acid was only slightly more effective than 10% acid, and that refluxing for one hour removes as much uranium as refluxing for four hours. Refluxing for one hour removed about 90% of the uranium.

One experiment conducted to determine the effectiveness of continuous leaching indicated that uranium was more completely removed in a shorter period of time by digestion than by continuous leaching.

Experiments were conducted in which simultaneous extraction and grinding were done. The ground carbon-parts and nitric acid were rotated in a porcelain ball mill, and it was determined that grinding during extraction with nitric acid has no appreciable effect upon the percentage of uranium extracted.

H. K. Jackson

HKJ:jcp

CC: M. McArdle (1)
H.K.Jackson (2)
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